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Wear 253 (2002) 1070–1076

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Tribological behaviour of Si_3N_4–BN ceramic materials for dry sliding applications

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Received 23 April 2002; received in revised form 17 July 2002; accepted 17 July 2002

Abstract

The main objective of this paper is to help on the clarification of the lack of consensus in the bibliographic data concerning the tribological behaviour of Si_3N_4–BN composites. Unlubricated sliding tests by pin-on-disc were carried out with three grades of composite materials with 10, 18 and 25 vol.% of BN. The addition of BN to the Si_3N_4 matrix resulted in a slight reduction of the friction coefficient, which decreased from 0.82 for monolithic Si_3N_4 to 0.67 for Si_3N_4–10%BN materials. Wear coefficients (K) were above $10^{-5}\,mm^3\,N^{-1}\,m^{-1}$ for all materials tested and increased sharply with increases in BN volume fraction greater than 10%, e.g. $K \sim 10^{-3}\,mm^3\,N^{-1}\,m^{-1}$ for Si_3N_4–25%BN. The lowest values of friction and wear coefficients were obtained when the composites were tested with the BN platelets oriented parallel to the sliding direction. The morphological study of the worn surfaces revealed generalised brittle intergranular microcracking at the dispersoid/matrix interface as the main wear mechanism. Under the experimental conditions of this study, the formation of stable protective layers of the soft lubricious oxide H_3BO_3, or the solid lubricant $BN{\cdot}H_2O$, was not observed.

Keywords: Tribological behaviour; Si_3N_4–BN composites; Silicon nitride; Dry sliding

1. Introduction

The demand for non- or boundary-lubricated tribosystems is increasingly a decisive design requirement in modern engineering applications, not only for ecological and economical reasons [1–3], but also by the call for the development of devices operating under extreme conditions (high temperature, vacuum) [4,5]. Si_3N_4-based ceramics are potential substitutes for more traditional materials for these specific applications due to their high hardness, excellent chemical and mechanical stability under a broad range of temperatures, low density, low thermal expansion and high specific stiffness [6]. The incorporation of solid lubricants is a goal to further enhance the tribological performance of Si_3N_4 [1,2,7–9]. Several Si_3N_4-based ceramic matrix composites (CMCs) have been tested for this purpose. Ceramic reinforcements (SiC, TiC, TiN, BN), in particulate or whisker forms, have been added in order to yield self-lubricating tribolayers by selective oxidation of these compounds, or simply to increase the fracture toughness of the ceramic body. However, results from distinct authors are somewhat contradictory. For example, the group of Woydt and co-workers [10–12] reported that the addition of 30 wt.% TiN did not significantly affect the Si_3N_4 wear coefficient at room temperature, but it enhance the composite wear resistance at elevated temperatures ($K \approx 10^{-5}\,mm^3\,N^{-1}\,m^{-1}$) up to $T = 800\,^{\circ}C$. At such temperature, the wear coefficient of the monolithic Si_3N_4 material is two orders of magnitude higher. The authors attributed the improvement in the wear resistance at elevated temperatures to the formation of TiO_{2-x} stable, wear resistant, oxides, and to the reduction of internal stresses by deformation of the TiN particles. Interestingly, the addition of TiN increased the friction coefficient, f, in almost all the set of experiments, including a relatively high value of $f = 1.2$ at $T = 800\,^{\circ}C$, for intermediate sliding speeds ($v = 0.3\,m\,s^{-1}$). The work of Melandri et al. [13] is somewhat contradictory to these results, as the tribological behaviour of TiN/Si_3N_4, and also of TiC/Si_3N_4 composites, showed no difference to the Si_3N_4 monolithic material in sliding experiments carried out from room temperature up to 1200 °C.

Concerning the addition of SiC whiskers, platelets or nanoparticles, several studies have indicated no net differences in the tribological response of the monolithic and

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PII: S0043-1648(02)00219-3

composite ceramics [14–16]. The easy debonding of SiC whiskers contributed to the degradation of the ceramic abrasive wear resistance [15,16]. Hot-pressed composites with SiC platelets parallel oriented to the sliding surface presented surplus surface damage by cleavage of the coarse SiC grains [14]. The present team has recently reviewed [14,17] the tribological behaviour of monolithic Si_3N_4 ceramics and Si_3N_4–SiC composites in unlubricated sliding contacts as self-mated pairs. For monolithic materials, a severe degradation of the Si_3N_4 wear resistance with temperature occurs, evidenced by increasing values of both wear and friction coefficients, from $K \approx 10^{-5}\,mm^3\,N^{-1}\,m^{-1}$ and $f = 0.2$ at room temperature to $K \geq 10^{-2}\,mm^3\,N^{-1}\,m^{-1}$ and $f \geq 1$ for experiments conducted at or above 1000 °C. At low temperatures, microcracking at the intergranular phase due to fatigue-assisted stresses prevails as dominant wear mode, while tribo-oxidation controls the materials degradation above 900 °C.

Hexagonal boron nitride is a well-known in situ lubricating material [8,10,11,18]. The formation of oxide or hydrated layers (H_3BO_3 and $BN{\cdot}(H_2O)_x$) is reported to have a beneficial effect on the tribological performance of Si_3N_4–BN composites as self-mated pairs at room temperature, reducing the wear coefficient one order of magnitude to $K \approx 10^{-6}\,mm^3\,N^{-1}\,m^{-1}$, relative to the matrix material [10]. The friction coefficient also decreases from $0.4 < f < 0.8$ for the matrix, to $0.1 < f < 0.4$ for the composite [11,18]. The self-lubricating effect of boric acid (H_3BO_3) is due to its layered triclinic crystal structure [19]. Both tribological parameters are reported to be almost independent of the sliding speed and BN content [11]. As temperature increases, the thin lubricant layers of $BN{\cdot}(H_2O)_x$ and H_3BO_3 are destroyed either by vaporisation of water, or by thermal decomposition above 150 °C. At elevated temperatures, the beneficial mechanism of tribo-oxidation/film formation changes to surface fatigue with extensive microcracking and delamination. Furthermore, BN starts to oxidise at 300 °C, hindering the lubricating role of this dispersoid. At 400 °C, the wear coefficient rises to a catastrophic value of $K > 10^{-4}\,mm^3\,N^{-1}\,m^{-1}$ [10]. A completely distinct behaviour is reported [20] for a specific application: a rotor seal working in an engine under fuel combustion products with an heat flow of $3000\,W\,m^{-2}\,K^{-1}$ at 1150 °C, at slow rotational speed between 70 and 100 r.p.m. Under these conditions, Si_3N_4–BN composites have shown a better performance than Si_3N_4 monolithic materials due to the formation of a passivating film with a complex chemical nature.

The tribological performance of Si_3N_4–BN composites has also been studied in sliding against a steel counterface [8]. Results reveal that the BN addition has no net effect on the friction and wear coefficients, an opposite trend of the above-cited works. In this case, boron nitride is reported to be ineffective in providing a solid lubricating film at room temperature. A comparison of the sliding wear behaviour of monolithic Si_3N_4 and Si_3N_4–BN composites in self-mated experiments, in the presence of water, revealed a slight increase in the wear coefficient with BN additions up to 70 wt.% [9]. The steady-state friction values showed no straight dependence on the BN content.

In the present work, the tribological behaviour of Si_3N_4–BN composites is further investigated to define the role of the dispersoid solid lubricant phase on the wear mechanisms. The influence of the BN platelets orientation is further discussed.

2. Materials and experimental details

Monolithic Si_3N_4 and BN–Si_3N_4 composite materials with 10, 18 and 25 vol.% BN were produced by uniaxial hot-pressing at 25 MPa of applied pressure in the temperature range 1450–1650 °C for 60 min of dwell time. The uniaxial hot-pressing technique allowed the fabrication of composite materials with BN platelets that were oriented either parallel (//) or perpendicular (⊥) to the sliding surface. Fabrication procedures and materials characterisation were detailed elsewhere [21,22]. Tribological tests were carried out in a controlled atmosphere at room temperature ($T = 22$ °C; RH = 50%). Tests were performed in a pin-on-disc tribometer (Plint TE67), without lubrication, at a constant sliding speed ($v = 2\,m\,s^{-1}$) and normal load ($W = 5$ N). The friction coefficient, f, is the ratio between the frictional force and the normal load. The wear coefficient, K, is given by $K = V/(W_x)$, where V represents the volumetric wear volume assessed by weight loss using a microbalance (accuracy = 10 μg), W is the normal load and x the sliding distance. The initial running-in period was not accounted for the calculation of f and K. Wear and friction coefficients were obtained from the average of values taken from 3 to 4 runs of 1 km of sliding distance each one. As an exception to this, the systems that presented an excessive loss of mass only allowed single runs of 300–500 m. The morphological analysis and chemical characterisation of the worn surfaces and wear debris were made by SEM/EDS and X-ray diffraction analysis.

3. Results

Fig. 1(a) shows the morphology of the hexagonal BN platelets used as reinforcement phase. Fig. 1(b) and (c) illustrate the typical microstructures of the Si_3N_4–BN composites, respectively with the BN platelets perpendicular or parallel oriented to the surface. In the last configuration, the basal planes of the platelets appear in their true dimension in the form of discs.

Fig. 2 presents the friction coefficients obtained in the experiments with the Si_3N_4–Si_3N_4 sliding pair and in the tribosystems involving the BN–Si_3N_4 composites as a function of BN volume fraction and BN platelet orientation in the pin. The addition of BN platelets to the Si_3N_4 matrix decreases the friction coefficient, this effect remaining

Fig. 1. SEM micrographs of (a) BN platelets; unworn surfaces of Si_3N_4–25 vol.% BN composites in perpendicular (b) and parallel (c) planes to the sliding motion.

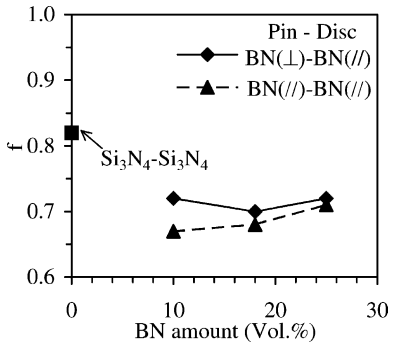

Fig. 2. Friction coefficient for self-mated and dissimilar tested couples with different BN amounts and platelet orientations (// and ⊥ correspond to parallel and perpendicular to the sliding surface, respectively).

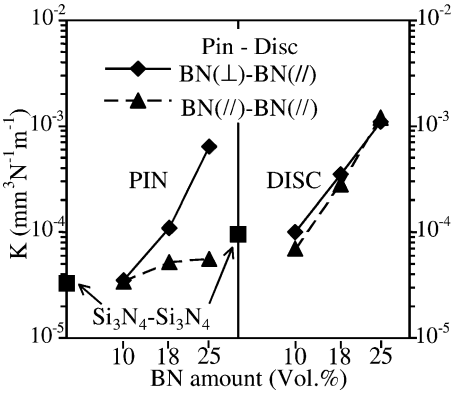

Fig. 3. Pin and disc wear coefficient for self-mated and dissimilar tested couples with different BN amounts and platelet orientations (// and ⊥ correspond to parallel and perpendicular to the sliding surface, respectively).

independent of the BN amount. Also, the friction values are always lower when the BN platelets are aligned parallel with the contact surface.

The complete set of wear coefficient values for the same experiments is depicted in Fig. 3. In view of the friction and wear behaviour, the best results were obtained for the Si_3N_4–10%BN(//) composite. BN additions higher than 10 vol.% are detrimental to the ceramic wear resistance, although the improvement in friction properties is maintained. Disc wear coefficients are always superior to the mating pin wear values. A notable feature is the effect of the BN platelets alignment on the wear characteristics, with the composite pins with BN oriented parallel to the sliding surface giving the best wear response.

The worn surface morphologies of the monolithic Si_3N_4 mating surfaces and the representative wear debris are illustrated in Fig. 4. The surface of the pin (Fig. 4(a)) reveals a combination of polished zones alternating with rough areas. The latter comprise adherent debris with a raft appearance, as evidenced in the right bottom corner of Fig. 4(a). This feature is particularly present on the disc worn track (Fig. 4(b)), where it provides evidence of a high degree of extensive fracture of both the bulk substrate and the debris tribolayer. The morphology of the corresponding loose wear debris is shown in Fig. 4(c), consisting of submicron particles. EDS analysis of the pin and disc worn surfaces and EDS and XRD analysis performed on loose wear debris do not indicate the presence of either silica or boron oxide.

The Si_3N_4–10%BN(⊥) composite pin surface after sliding on the Si_3N_4–10%BN(//) disc (Fig. 5(a)) is similar in appearance to that of the monolithic material, being partially covered by adherent debris layers. This debris layer covers the perpendicularly oriented BN platelets that present a prismatic-shape geometry in a polished unworn surface, as shown in a previous work [22]. Otherwise, when the BN platelets are parallel to the sliding surface, as documented in Fig. 5(b), they reveal a disc-shaped configuration in the polished area of the Si_3N_4–10%BN(//). The same morphology

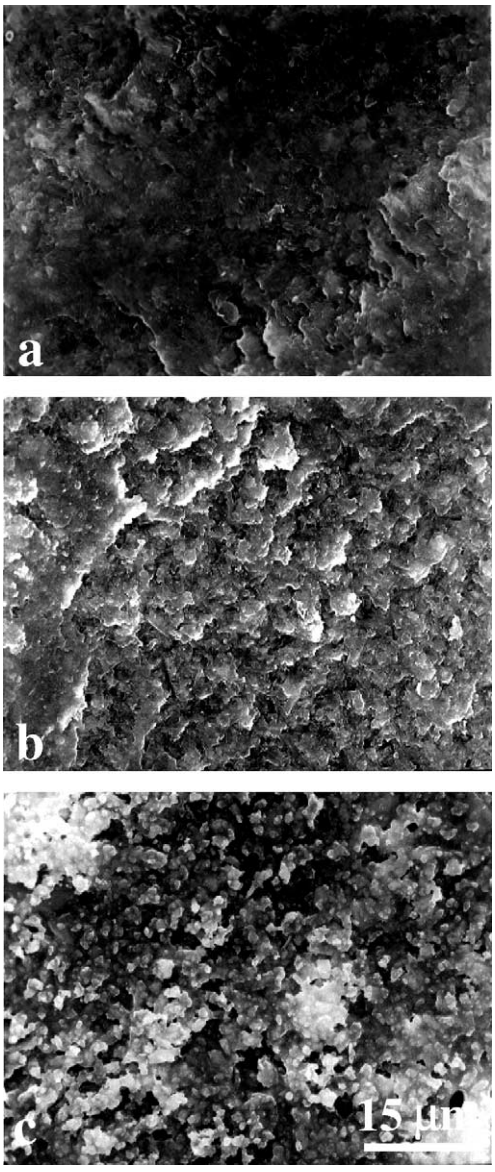

Fig. 4. SEM images after Si_3N_4/Si_3N_4 sliding experiments: (a) Si_3N_4 pin surface; (b) Si_3N_4 disc surface; (c) loose wear debris morphology.

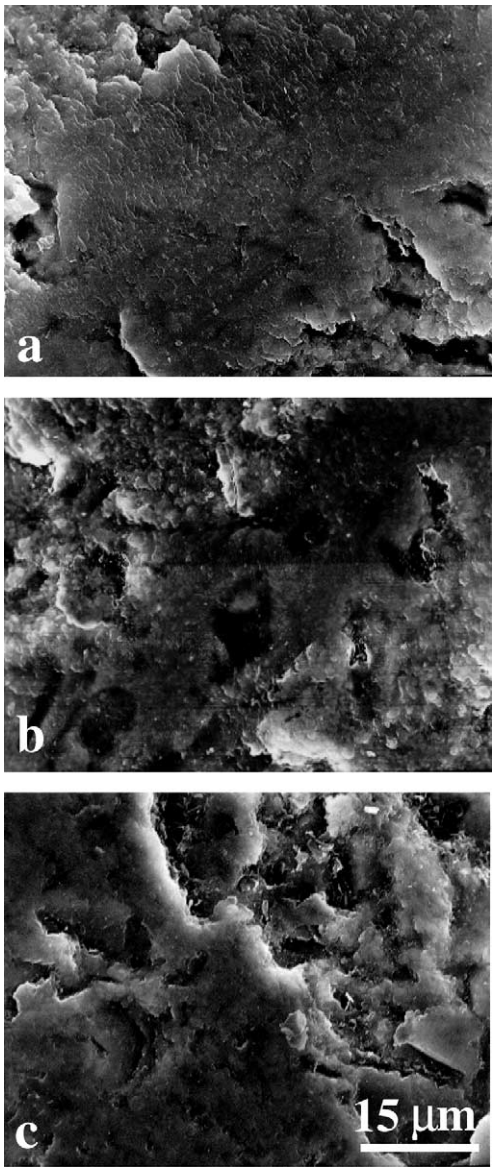

Fig. 5. SEM images after Si_3N_4–10%BN/Si_3N_4–10%BN sliding experiments: (a) pin surface with perpendicular BN platelets; (b) pin surface with parallel BN platelets; (c) disc surface with parallel BN platelets.

is observed in the corresponding disc (Fig. 5(c)), although with a high level of material spalling.

For the ceramic composites with BN volume fractions greater than 10%, BN platelet orientation is an important variable in defining wear resistance, as illustrated in Fig. 3. The Si_3N_4–25%BN(⊥) pin damaged morphology in Fig. 6(a) is a consequence of the catastrophic failure of the composite. On the contrary, when the dispersoid phase is parallel oriented to the sliding motion (Fig. 6(b)), the composite shows a much more preserved and smooth surface, denoting an extensive coverage by a aggregated tribolayer with a few cohesive cracks. The Si_3N_4–25%BN(//) disc is characterised by the highest wear coefficient (Fig. 3) as corroborated by the fractured surface in Fig. 6(c). Some deep cavities corresponding to the initial location of BN platelets are visible. The representative morphology of the loose wear debris originated from this tribopair are composed by a mixture of submicron particles and large agglomerates (>10 μm) as shown in Fig. 6(d).

4. Discussion

The tribological characteristics of monolithic Si_3N_4 ceramics sliding against themselves is a well-known matter [10–12,17,23]. In the present test conditions, the ceramic wear resistance is low, with friction and wear coefficients unacceptable for practical applications, i.e., higher than 0.5 and 10^{-5} $mm^3\,N^{-1}\,m^{-1}$, respectively, see Figs. 2 and 3. The

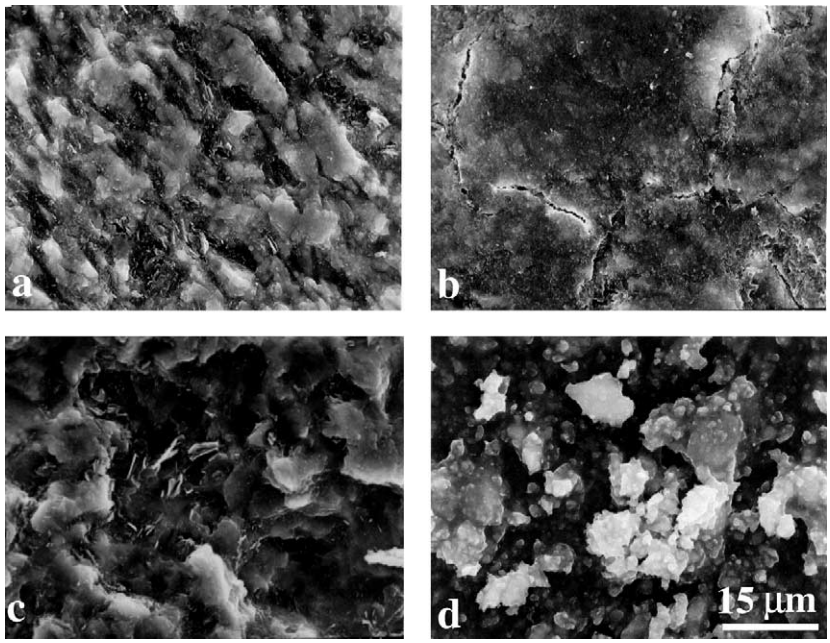

Fig. 6. SEM images after Si_3N_4–25%BN/Si_3N_4–25%BN sliding experiments: (a) pin surface with perpendicular BN platelets; (b) pin surface with parallel BN platelets; (c) disc surface with parallel BN platelets; (d) representative loose wear debris morphology.

dominant wear mechanism is the mechanical failure of the material, as denoted by the wear pattern of the mating surfaces, see Fig. 4(a) and (b). Subsurface cracks propagate towards the surface assisted by fatigue effects leading to fractured rough regions, combined with small polished areas from fine scale abrasion [10,14,23,24]. Also, the resultant large amount of Si_3N_4 wear debris (Fig. 4(c)) tends to clump on the ceramics surface contributing to the rough appearance of the contact surface. Compared to the pins, the Si_3N_4 discs show a higher wear loss (Fig. 3) and surface damage (Fig. 4(b)), due to intermittent loading [4]. In this study, no reactive processes like tribo-oxidation were identified. Thus, no chemical processes were activated at the contact interface to produce any kind of oxide layer that may affect the tribological behaviour. As a result, wear behaviour is entirely determined by the mechanical response of the ceramic surfaces. The absence of tribochemical phenomena is attributed to the relatively moderate test conditions, i.e., room temperature, moderate relative humidity, low load and sliding speed, which were insufficient to activate chemical reactions between the mating surfaces and the surrounding atmosphere.

The decrease of the friction coefficient with addition of BN particles (Fig. 2) is in agreement with literature results obtained with Si_3N_4–20%BN composites, for similar conditions of load, temperature and sliding speed [11] and Si_3N_4–47.5%BN materials tested at lower speeds [18]. This decrease in friction coefficient is typically attributed to BN hydration, which provides an in situ lubrication [8,10,18]. However, the hydration of BN was not observed in the present study, and the lower friction values obtained when the BN platelets are oriented parallel to the sliding surface (Fig. 2) are due to the easy cleavage of the basal planes of the hexagonal BN particles. For BN volumes greater than 10 vol.%, the friction coefficient tends to increase due to the overlapping of the mechanical component of friction, given by the rougher surfaces (Fig. 6(a) and (c)) over the lubricating action. Moreover, a lack of hydrated lubricating material is attributed to both ineffective BN supply and poor BN adhesion to the surface [8].

The positive effect of BN on friction is accompanied by an increase in wear resistance only when low BN amounts (10 vol.%) are added to the Si_3N_4 matrix and the BN platelets are oriented parallel to the surface, see Fig. 3. These composites are characterised by hardness values in the range 12–14.5 GPa [22] and average fracture toughness values of 5.8 MPa $m^{1/2}$ [22]. For intermediate (18 vol.%) and high (25 vol.%) BN contents, the hardness decreases significantly to 8 and 5 GPa, respectively. The K_{IC} also decreases down to 4.6 MPa $m^{1/2}$ [22]. The incorporation of BN as a solid lubricant in the ceramic matrix weakens the material's cohesion and its mechanical strength [2]. Hence, the loss of mechanical resistance governs the degradation of the wear properties of such composites. For this reason, our results are in contradiction with that reported for a Si_3N_4–20 wt.%BN composite with an extremely low hardness value of 2 GPa [10,11] that presented a wear coefficient of 10^{-6} mm^3 N^{-1} m^{-1}, one order of magnitude lower than that of the monolithic Si_3N_4 material. However, this remarkable result seemed to be not further surveyed. The data from our experiments support the opposite trend as obtained by other authors, that report the ineffectiveness of BN to significantly improve the Si_3N_4 wear properties [8,9].

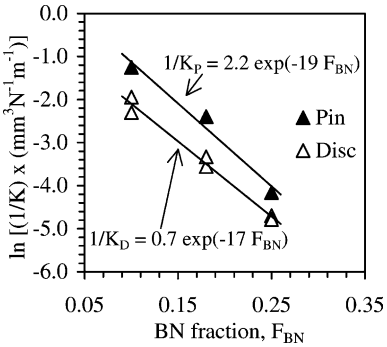

Fig. 7. Semi-logarithmic dependence of the wear resistance (1/*K*) vs the volume fraction of BN dispersoid phase of the Si_3N_4–BN composites.

The wear resistance of the present ceramic composites, 1/*K*, shows a semi-logarithmic dependence on the BN volume fraction, F_{BN}, as depicted in Fig. 7. As reported above, the BN platelets tend to detach from the Si_3N_4 matrix during the sliding motion. Fig. 8 shows the high magnification image of a cavity with the characteristic dimensions of a BN platelet (Fig. 1(a)) denoting that interfacial failure between BN dispersoids and Si_3N_4 matrix is a primary source of material loss during the tribological action. Thus, porous zones arise at the contact surface leading to a loss on the mechanical properties of the composite material. This explains the linear trend in Fig. 7 that is in accordance with the universal Knudsen's type exponential dependence on porosity [25]. This model is based on the empirical fitting of the ceramic materials mechanical properties dependence on porosity by an equation of the type $MP = A\exp(-bP)$, where MP is the value of a generic "mechanical property", *A* is a constant and *b* is the porosity (*P*) factor. Several properties follow this trend, like Young's and other elastic modulus, hardness, compressive strength, etc., which is explained by the lack of resistive volume linked to the presence of porosity [25]. The *y*-intercept value is lower for the disc line as a consequence of the intense cyclic thermomechanical loading on the disc [10]. The pronounced negative slope in Fig. 7 points out the prevalence of the microstructural characteristics of the ceramic composites on their wear resistance, instead of any lubricious protective effect from BN additions.

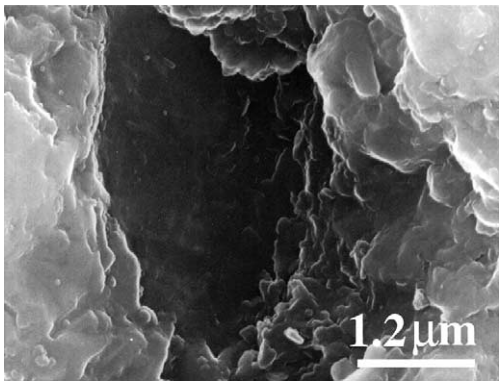

Fig. 8. High magnification SEM image of a cavity at the Si_3N_4–BN composite surface corresponding to a BN platelet detachment.

5. Conclusions

Silicon nitride materials profit from the addition of boron nitride platelets to improve their tribological properties only if low volume amounts (10%) are incorporated. An improvement in wear resistance occurs when the BN platelets have their easy cleavage basal planes oriented parallel to the sliding surface, affording a lubricious effect. No BN hydration was detected in this study, most likely due to the moderate test conditions, which were insufficient to activate such chemical reactions with the surrounding environment.

For BN contents greater than 10 vol.%, the mechanical properties of the Si_3N_4–BN composites decrease significantly due to the very soft nature of BN and the brittle response of the dispersoid/matrix interface, leading to unacceptable high friction and wear coefficients. The wear response is controlled by fatigue-assisted generalised surface microcracking due to the lack of mechanical resistance of the composites. An exponential dependence of the wear resistance on the BN volume fraction was found. This feature is analogous to the Knudsen's type dependence on porosity. For the present composites, porous zones outcome from the BN easy detachment during sliding motion corroborating this model.

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